### CETIFICATION

SDG No:

MC45838

Humacao, PR

Laboratory:

Accutest, Massachusetts

Site:

BMS, Building 5 Area, PR

Matrix:

Groundwater

**SUMMARY:** 

Groundwater samples (Table 1) were collected on the BMSMC facility – Building 5 Area. The BMSMC facility is located in Humacao, PR. Samples were taken May 9-10, 2016 and were analyzed in Accutest Laboratory of Marlborough, Massachusetts that reported the data under SDG No.: MC45838. Results were validated using the following quality control criteria of the methods employed (MADEP VPH and MAPED EPH, Massachusets Department of Environmental Protection, 2004) and the latest validation guidelines (July, 2015) of the EPA Hazardous Waste Support Section. The analyses performed are shown in Table 1. Individual data review worksheets are enclosed for each target analyte group. The data sample organic data samples summary form shows for analytes results that were qualified.

In summary the results are valid and can be used for decision taking purposes.

Table 1. Samples analyzed and analysis performed

SAMPLE ID	SAMPLE DESCRIPTION	MATRIX	ANALYSIS PERFORMED
MC45838-1	S-35D	Groundwater	Volatiles TPHC Ranges
MC45838-1A	S-35D	Groundwater	Extractable TPHC Ranges
MC45838-3	S-41D	Groundwater	Volatiles TPHC Ranges
MC45838-3A	S-41D	Groundwater	Extractable TPHC Ranges
MC45838-4	S-42D	Groundwater	Volatiles TPHC Ranges
MC45838-4A	S-42D	Groundwater	Extractable TPHC Ranges
MC45838-5	RA6-GWS	Groundwater	Volatiles TPHC Ranges
MC45838-6	RA6-GWS	Groundwater	Extractable TPHC Ranges
MC45838-7	RA20-GWS	Groundwater	Volatiles TPHC Ranges
MC45838-7A	RA20-GWS	Groundwater	Extractable TPHC Ranges

Reviewer Name:

Rafael Infante

Chemist License 1888

Signature:

Date:

May 31, 2016

# Report of Analysis

Page I of 1

Client Sample ID:

S-35D MC45838-1

Lab Sample ID:

AQ - Ground Water

Matrix: Method:

MADEP VPH REV 1.1

Project:

BMSMC, Building 5 Area, Puerto Rico

Date Sampled:

05/09/16 05/11/16

Date Received:

Percent Solids:

n/a

Run #1	File ID AB94027.D	<b>DF</b>	Analyzed 05/11/16	By DF	Prep Date	Prep Batch	Analytical Batch GAB5167
Run #2		-	00711710	23.	11.4	Ша	GADJIOI

Purge Volume

Run #1

 $5.0 \, ml$ 

Run #2

# Volatile TPHC Ranges

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.) C9- C12 Aliphatics (Unadj.) C9- C10 Aromatics (Unadj.) C5- C8 Aliphatics C9- C12 Aliphatics	ND ND ND ND ND	50 50 50 50 50	40 40 40 40 40	ug/l ug/l ug/l ug/l	
CAS No.	Surrogate Recoveries  2,3,4-Trifluorotoluene	Run# 1	Run# 2	Lim	ug/l its 30%	
	2,3,4-Trifluorotoluene	86%			30%	





MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

# Report of Analysis

TA

05/12/16

Page 1 of 1

**Analytical Batch** 

**GDE791** 

Client Samp	le ID:	S-35D
-------------	--------	-------

Lab Sample ID:

MC45838-1A

Matrix: Method:

Project:

AQ - Ground Water

1

MADEP EPH REV 1.1 SW846 3510C BMSMC, Building 5 Area, Puerto Rico

Date Sampled:

05/09/16 Date Received: 05/11/16

Percent Solids: n/a

**OP47446** 

File ID DF Analyzed By Prep Date Prep Batch Run #1 DE14095.D

05/16/16 Run #2

Initial Volume **Final Volume** 

Run #1 Run #2

 $2.0 \, \mathrm{ml}$ 

# Extractable TPHC Ranges

960 ml

CAS No. Compound Result RL MDL Units	Q
C11-C22 Aromatics (Unadj.) ND 100 73 ug/l	
C9-C18 Aliphatics ND 100 73 ug/l	
C19-C36 Aliphatics ND 100 73 ug/l	
C11-C22 Aromatics ND 100 73 ug/l	
CAS No. Surrogate Recoveries Run#1 Run#2 Limits	
84-15-1 o-Terphenyl 78% 40-140%	
321-60-8 2-Fluorobiphenyl 77% 40-140%	
3386-33-2 1-Chlorooctadecane 67% 40-140%	
580-13-2 2-Bromonaphthalene 79% 40-140%	





MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

# Report of Analysis

DF

n/a

Page 1 of 1

Client Sample ID: S-41D

Lab Sample ID:

MC45838-3

Matrix: Method:

AQ - Ground Water

Project:

MADEP VPH REV 1.1

DF

1

BMSMC, Building 5 Area, Puerto Rico

Analyzed

05/11/16

Date Sampled: 05/10/16

Date Received: 05/11/16

Percent Solids: n/a

GAB5167

Ву	Prep Date	Prep Batch	Analytical Batch

n/a

Run #1 Run #2

Purge Volume

Run #1

 $5.0 \, ml$ 

File ID

AB94028.D

Run #2

# Volatile TPHC Ranges

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	50	40	ug/l	
	C9- C12 Aliphatics (Unadj.)	ND	50	40	ug/l	
	C9- C10 Aromatics (Unadj.)	ND	50	40	ug/l	
	C5- C8 Aliphatics	ND	50	40	ug/l	
	C9- C12 Aliphatics	ND	50	40	ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
	2,3,4-Trifluorotoluene	84%		70-1	30%	
	2,3,4-Trifluorotoluene	86%		70-1	30%	



# Report of Analysis

Page 1 of 1

Client Sample	ID: S	S-41D
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Lab Sample ID:

MC45838-3A

Matrix:

AQ - Ground Water

Date Sampled:

05/10/16

Method:

MADEP EPH REV 1.1 SW846 3510C

Date Received: 05/11/16

Project:

BMSMC, Building 5 Area, Puerto Rico

Percent Solids: n/a

File ID Ву DF Analyzed Prep Date Prep Batch **Analytical Batch** Run #1 DE14096.D 1 05/16/16 TA 05/12/16 OP47446 **GDE791** 

Run #2

**Initial Volume Final Volume** 

Run #1

945 ml 2.0 ml

Run #2

# **Extractable TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.) C9-C18 Aliphatics C19-C36 Aliphatics C11-C22 Aromatics	ND ND ND ND	110 110 110 110	74 74 74 74	ug/l ug/l ug/l ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
84-15-1 321-60-8 3386-33-2 580-13-2	o-Terphenyl 2-Fluorobiphenyl 1-Chlorooctadecane 2-Bromonaphthalene	79% 77% 57% 79%	40-140% 40-140% 40-140% 40-140%		40% 40%	



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

# Report of Analysis

Page 1 of 1

Client Sample ID: Lab Sample ID:

S-42D MC45838-4

Matrix:

Method:

AQ - Ground Water

MADEP VPH REV 1.1

BMSMC, Building 5 Area, Puerto Rico

Date Sampled: Date Received:

05/10/16 05/11/16

Percent Solids: n/a

Project: DF Ву

File ID Run #1

AB94029.D

Analyzed 05/11/16 DF Prep Date n/a

Prep Batch n/a

**Analytical Batch** GAB5167

Run #2

Purge Volume

Run #1 Run #2  $5.0 \, \mathrm{ml}$ 

# Volatile TPHC Ranges

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	50	40	ug/l	
	C9- C12 Aliphatics (Unadj.)	ND	50	40	ug/l	
	C9- C10 Aromatics (Unadj.)	ND	50	40	ug/l	
	C5- C8 Aliphatics	ND	50	40	ug/l	
	C9- C12 Aliphatics	ND	50	40	ug/l	
CAS No.	Surrogate Recoveries	Run#1	Run# 2	2 Limits		
	2,3,4-Trifluorotoluene	83%		70-1	30%	
	2,3,4-Trifluorotoluene	86%		70-1	30%	



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

# Report of Analysis

Page 1 of 1

Client	Sample ID:	S-42D
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File ID

DE14097.D

Lab Sample ID:

MC45838-4A

Matrix:

AQ - Ground Water

DF

MADEP EPH REV 1.1 SW846 3510C

Date Sampled: Date Received:

05/10/16

Percent Solids:

05/11/16 n/a

Method: Project:

BMSMC, Building 5 Area, Puerto Rico

Analyzed By Prep Date Prep Batch **Analytical Batch** 05/16/16 TA 05/12/16 OP47446 **GDE791** 

Run #1 Run #2

Initial Volume

Final Volume

Run #1 Run #2 925 ml

 $2.0 \, ml$ 

# **Extractable TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.) C9-C18 Aliphatics C19-C36 Aliphatics C11-C22 Aromatics	ND ND ND ND	110 110 110 110	76 76 76 76	ug/l ug/l ug/l ug/l	
CAS No.	Surrogate Recoveries	Run#1	Run# 2	2 Limits		
84-15-1 321-60-8 3386-33-2 580-13-2	o-Terphenyl 2-Fluorobiphenyl 1-Chlorooctadecane 2-Bromonaphthalene	90% 91% 74% 93%	40-140% 40-140% 40-140% 40-140%		40% 40%	



ND = Not detected

MDL = Method Detection Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

# Report of Analysis

DF

Page 1 of 1

Client Sample ID:

**RA6-GWS** 

Lab Sample ID:

MC45838-5

Matrix: Method:

AQ - Ground Water

Project:

MADEP VPH REV 1.1 BMSMC, Building 5 Area, Puerto Rico Date Sampled:

05/09/16

Date Received:

05/11/16

Percent Solids:

n/a

			<del>-</del>		
] 1	File ID	DF	Analy	zed By	

1

Run #1 Run #2 Analyzed Ву 05/11/16

Prep Date n/a

Prep Batch

**Analytical Batch** 

n/a GAB5167

Purge Volume

AB94030.D

Run #1

 $5.0 \, ml$ 

Run #2

Volatile TPHC Ranges

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	50	40	ug/l	
	C9- C12 Aliphatics (Unadj.)	123	50	40	ug/l	
	C9- C10 Aromatics (Unadj.)	73.5	50	40	ug/l	
	C5- C8 Aliphatics	ND	50	40	ug/l	
	C9- C12 Aliphatics	ND	50	40	ug/I	
CAS No.	Surrogate Recoveries	Run#1	Run# 2	un#2 Limits		
	2,3,4-Trifluorotoluene	85%		70-1	30%	
	2,3,4-Trifluorotoluene	88%			30%	



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

# Report of Analysis

Page 1 of 1

	nt Sample III:
It ah	Comple II)

**RA6-GWS** MC45838-6

AQ - Ground Water

Date Sampled:

05/10/16

Matrix: Method:

Date Received: 05/11/16

Project:

MADEP EPH REV 1.1 SW846 3510C BMSMC, Building 5 Area, Puerto Rico

Percent Solids: n/a

	File ID	DF	Analyzed	Ву	Prep Date	Prep Batch	Analytical Batch
Run #1	DE14098.D	1	05/16/16	TA	05/12/16	OP47446	GDE791

|Run #2

Initial Volume Final Volume 780 ml

Run #1

2.0 ml

Run #2

# **Extractable TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.) C9-C18 Aliphatics C19-C36 Aliphatics C11-C22 Aromatics	93.6 ND ND 90.9	130 130 130 130	90 90 90 90	ug/l ug/l ug/l ug/l	1
CAS No.	Surrogate Recoveries	Run#1	Run# 2	2 Limits		
84-15-1 321-60-8 3386-33-2 580-13-2	o-Terphenyl 2-Fluorobiphenyl 1-Chlorooctadecane 2-Bromonaphthalene	77% 86% 58% 87%	40-140% 40-140% 40-140% 40-140%			



ND = Not detected

MDL = Method Detection Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

# Report of Analysis

By

DF

Page 1 of 1

Client Sample ID: RA20-GWS Lab Sample ID: MC45838-7

Matrix: Method: AQ - Ground Water

MADEP VPH REV 1.1

Date Sampled: 05/10/16 Date Received: 05/11/16

Percent Solids: n/a

Project: BMSMC, Building 5 Area, Puerto Rico

File ID Run #1

DF AB94031.D

Analyzed 05/11/16

Prep Date n/a

Prep Batch

**Analytical Batch** 

n/a GAB5167

Run #2

Purge Volume

Run #1 Run #2  $5.0 \, ml$ 

# Volatile TPHC Ranges

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	50	40	ug/l	
	C9- C12 Aliphatics (Unadj.)	ND	50	40	ug/l	
	C9- C10 Aromatics (Unadj.)	ND	50	40	ug/l	
	C5- C8 Aliphatics	ND	50	40	ug/l	
	C9- C12 Aliphatics	ND	50	40	ug/l	
CAS No.	Surrogate Recoveries	Run#1	Run# 2	Lim	its	
	2,3,4-Trifluorotoluene	85%		70-1	30%	
	2,3,4-Trifluorotoluene	87%		70-1	30%	



ND = Not detected

MDL = Method Detection Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

# Report of Analysis

Page 1 of 1

Client Sample ID: Lab Sample ID:

RA20-GWS MC45838-7A

Matrix:

AQ - Ground Water

MADEP EPH REV 1.1 SW846 3510C BMSMC, Building 5 Area, Puerto Rico

Date Sampled: 05/10/16 Date Received: 05/11/16

Percent Solids: n/a

File ID Run #1 DE14100.D

DF

Analyzed 05/16/16

By Prep Date TA 05/12/16

Prep Batch OP47446

**Analytical Batch GDE791** 

Run #2

Method:

Project:

Initial Volume

1-Chlorooctadecane

2-Bromonaphthalene

880 ml

**Final Volume**  $2.0 \, \mathrm{ml}$ 

Run #1 Run #2

3386-33-2

580-13-2

**Extractable TPHC Ranges** 

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	110	80	ug/l	
	C9-C18 Aliphatics	ND	110	80	ug/l	
	C19-C36 Aliphatics	ND	110	80	ug/l	
	C11-C22 Aromatics	ND	110	80	ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
84-15-1	o-Terphenyl	74%		40-1	40%	
321-60-8	2-Fluorobiphenyl	81%		40-I	40%	

56%

83%



40-140%

40-140%

ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

SGS	Andrew Control of the	-NE TEL 732-3	SGS Accutes of	CUST No.	ODY SIE	ngla Di	nd Angelo IN M/	38			•		MU	400
Anderson Mulholand	Zo Crow Project 8	SMS Relea		5505	SS (M	CN	<u>}</u>	HAN	EPHR					Metric, Codes  DW - Drivers Wah  DW - Creard Wah  DW - Creard Wah  SW - Serfice Web  BO - Ser  SL - Steiger  SU - Serfice Steiner  CS - Ce  LO - Chre Labed  ART - Ar  BO - Who  FB - Finit Shee  CR - Ce  SE - CE
has Field D / Point of Collection/ 5 r 35 D	NEONOT Vist #	on 1m	NR 6W		10 at	POSE NO	INCOM	N X	BMA					LAB USE ONLY
-2 RA-20 (6.5		5/10/16 HSD 5/10/16 HAB	TT SO R. GW	5			3	X X X	XXX					
-Y S-42D -F RAG-6WS -G RAG-GW -7 RA20-GW	5 5 1.5	5/9/16 1645	TT GW	3 3	-			X	X					1017
														17A 361
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MC45838: Chain of Custody Page 1 of 2

#### **EXECUTIVE NARRATIVE**

SDG No:

MC45838

Laboratory:

**Accutest, Massachusetts** 

Analysis:

**MADEP VPH** 

Number of Samples:

Location:

BMSMC, Building 5 Area

Humacao, PR

SUMMARY:

Five (5) samples were analyzed for Volatiles TPHC Ranges by method MADEP VPH. Samples were validated following the METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (VPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the

primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

Critical issues:

None

Major:

None

Minor:

None

**Critical findings:** 

None

Major findings:

None

Minor findings:

% differences in the rt5.5-7 hydrocarbon range did not meet the method and guidance document performance criteria in the initial calibration verification. Results qualified as estimated (J) or (UJ) in affected samples (JC45838-1; -3; -4; -5; -7).

COMMENTS:

Results are valid and can be used for decision making purposes.

Reviewers Name:

Rafael Infante

Chemist License 1888

Signature:

Date:

May 31, 2016

# SAMPLE ORGANIC DATA SAMPLE SUMMARY

Sample ID: MC45838-1

Sample location: BMSMC Building 5 Area Sampling date: 5/9/2016

Matrix: Groundwater

# METHOD: MADEP VPH

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
50	50	50	50	50	Result
ug/L	ug/L	ug/L	ug/L	ug/L	Units Dilu
⊢	1	<u>⊢-</u>	ш	↦	Dilution Factor 1
,	•		ı	•	Lab Flag
<b>C</b>	<b>C</b>	<b>–</b>	<b>C</b>	٥	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

Sample ID: MC45838-3

Sample location: BMSMC Building 5 Area

Sampling date: 5/10/2016

Matrix: Groundwater

# METHOD: MADEP VPH

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
50	50	50	50	50	Result
ug/L 1	ug/L 1	ug/L 1	ug/L 1	ug/L 1	Units Dilution Factor
1		1		•	Lab Flag 1
<b>C</b>	<b>C</b>	<b>C</b>	<b>C</b>	⊆	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

Sample ID: MC45838-4

Sample location: BMSMC Building 5 Area

Sampling date: 5/10/2016

Matrix: Groundwater

# METHOD: MADEP VPH

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
50	50	50	50	50	Result
ug/L 1	ug/L 1	ug/L 1	ug/L 1	ug/L 1	<b>Units Dilution Factor</b>
1	•	•	•	1	Lab Flag
<b>C</b>	<b>C</b>	<b>C</b>	c	٤	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

Sample ID: MC45838-5

Sample location: BMSMC Building 5 Area

Sampling date: 5/9/2016

Matrix: Groundwater

# METHOD: MADEP VPH

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
50	50	73.5	123	50	Result
ug/L 1	ug/L 1	ug/L 1	ug/L 1	ug/L 1	Units Dilution Factor
1	t	·	•	•	Lab Flag
_	C		•	٤	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

Sample ID: MC45838-7
Sample location: BMSMC Building 5 Area
Sampling date: 5/10/2016
Matrix: Groundwater

# METHOD: MADEP VPH

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
50	50	50	50	50	Result
ug/L 1	ug/L 1	ug/L 1	ug/L 1	ug/L 1	Units Dilution Factor Lab Flag Validation Reportabl
				•	Factor L
ī	ı	1	,	•	ab Flag
C	<b>C</b>	1		⋸	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

# **DATA REVIEW WORKSHEETS**

Type of validation	Full:X Limited:	Project Number: _MC45838
REVIEW OF VO	DLATILE PETROLEUI	M HYDROCARBON (VPHs) PACKAGE
validation actions. This of more informed decision were assessed according precedence METHOD HYDROCARBONS (VPI (2004). Also the general	document will assist the and in better serving to the data validation FOR THE DETE  H), Massachusetts Depart validation guidelines  C criteria and data validation	le organics were created to delineate required reviewer in using professional judgment to make the needs of the data users. The sample results an guidance documents in the following order of RMINATION OF VOLATILE PETROLEUM artment of Environmental Protection, Revision 1.1 promulgated by the USEPA Hazardous Wastes ation actions listed on the data review worksheets to otherwise noted.
The hardcopied (labor received has been review review for SVOCs included)	atory name) _Accutes wed and the quality con ed:	t_Laboratories data package trol and performance data summarized. The data
No. of Samples: Field blank No.: Equipment blank No.:	5 - - -	Sample matrix:Groundwater
X Data CompleteX Holding TimesN/A GC/MS TuningN/A Internal StandaX BlanksX Surrogate Reco	rd Performance	X Laboratory Control SpikesX Field DuplicatesX CalibrationsX Compound IdentificationsX Compound QuantitationX Quantitation Limits
Overall Comme (C5_to_C12_Aliphatics;_	nts: _Volatiles_ C9_to_C10_Aromatics)	by_GC_by_Method_MADEP_VPH,_REV_1.1
Definition of Qualifiers:		
J- Estimated results U- Compound not de R- Rejected data UJ- Estimated nonde  Reviewer: 4/46 Date: 05/31/2016	etected	

	Criteria were not n	All criteria were metx net and/or see below
I. DATA COMPLETNE A. Data Packag		
MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
3. Other		Discrepancies:

All criteria were met	_X
Criteria were not met and/or see below	

#### **HOLDING TIMES**

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED	DATE ANALYZED	ACTION
Sa	amples analyzed	within method re	commended holdi	ng time

### Criteria

### Preservation:

Samples analyzed with ambient purge temperature: Samples must be acidified to a pH of 2.0 or less at the time of collection.

Samples analyzed with heated purge temperature: Samples must be treated to a pH of 11.0 or greater at the time of collection.

Methanol preservation of soil/sediment samples is mandatory. Methanol (purge-and-trap grade) must be added to the sample vial before or immediately after sample collection. In lieu of the in-field preservation of samples with methanol, soil samples may be obtained in specially-designed air tight sampling devices, provided that the samples are extruded and preserved in methanol within 48 hours of collection.

# Holding times:

Aqueous samples using ambient or heated purge - analyze within 14 days. Soil/sediment samples - analysis within 28 days.

Cooler temperature	(Criteria:	4 <u>+</u> 2 '	°C):	4.8°C_	
--------------------	------------	----------------	------	--------	--

Actions: Qualify positive results/nondetects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ). If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R). If samples were not at the proper temperature (> 10°C) or improperly preserved, use professional judgment to qualify the results.

	All cr Criteria were not met a	iteria were met and/or see below _	
CALIBRATIONS VERIFICATION			
Compliance requirements for sa ensure that the instrument is quantitative data.	atisfactory instrument calibratic capable of producing and	tion are establish maintaining acce	ed to ptable
	Date of initial calibration:	01/12/16	

Date of initial calil	oration:	_01/12/16_	
Dates of initial cal	ibration v	rerification:_	_01/12/16_
Instrument ID nur	nbers:	GCA	B
Matrix/Level:	AQUE	OUS/MEDIL	IM

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, <u>%D</u> , r	SAMPLES AFFECTED
01/12/16	icv5058-50	rt5.5-7	22.6	MC45838-1; -3; -4; -5; -7

Note: Initial and initial calibration verification meet method specific requirements except in the cases described in this document.

#### Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of interest. Calculate the collective CFs for C5-C8 Aliphatic Hydrocarbons and C9-C12 Aliphatic Hydrocarbons using the FID chromatogram. Calculate the collective CF for the C9-C10 Aromatic Hydrocarbons using the PID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest.

Criteria- CCAL

# DATA REVIEW WORKSHEETS

- At a minimum, the working calibration factor must be verified on each working day, after every 20 samples, and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.
- If the percent difference (%D) for any analyte varies from the predicted response by more than ±25%, a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

#### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects. If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

#### **CALIBRATIONS VERIFICATION**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration.	_01/12/16				
Dates of continuing calibration vo	erification:05/11/16				
Dates of final calibration verification:05/12/16					
Instrument ID numbers:	_GCAB				
Matrix/Level:AQUEOUS/N	IEDIUM				

DATE	LAB FILE	ANALYTE	CRITERIA OUT	SAMPLES
	LID#		RFs, %RSD, <u>%D,</u> r	AFFECTED
Instrument	: GCBH			
05/11/16	cc5058-50	rt5.5-7	39.6	MC45838-1; -3; -4;
05/11/16	cc5058-50	rt5.5-7	43.9	-5; -7
			<del></del>	
	4.5			

Note: Continuing and final calibration verification meet method specific requirements except for the case described above. Results for hydrocarbons in this range above reporting limits are qualified as estimated (J) non-detects are qualified (UJ).

A separate worksheet should be filled for each initial curve

				All culteria were tiletX
			Criteria were not	met and/or see below
VA. BLANK	ANALYSIS R	ESULTS (Se	ctions 1 & 2)	
magnitude of complete blanks associate problems with evaluated to decase, or if the problems with the	ontamination ed with the s any blanks e termine whet problem is an must be run	problems. The samples, included in the samples, included in the sample after sample after sample in the sample in the sample after sample in the sample in t	ne criteria for eva auding trip, equipm a associated with ere is an inheren currence not affect as suspected of	letermine the existence and luation of blanks apply only to nent, and laboratory blanks. In the case must be carefully t variability in the data for the cting other data. A Laboratory being highly contaminated to
List the contami separately.	ination in the	e blanks belo	w. High and low	levels blanks must be treated
Laboratory blank	ks			
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
METHOD BL			<del></del>	ITERIA
Field/Trip/Equip	ment			
A methanol trip I each soil/sedim storage, and ana	ent sample	ified reagent of water sa	water trip blank si ample batch, re	hould continually accompany spectively, during sampling,
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_PACKAGE	·			WITH_THIS_DATA

All criteria were met	X
Criteria were not met and/or see below	

# V B. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is < sample quantitation limit (SQL) and < AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but < AL, report the compound as not detected (U) at the reported concentration.

If the concentration is > AL, report the concentration unqualified.

SAMPLE ID

All criteria were met _	X
Criteria were not met and/or see below	

ACTION

# SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery. Matrix: solid/aqueous

SURROGATE COMPOUND

2,3,4	1-Trifluorotoluene		•	
_SURROGATE_STAN	IDARD_RECOVERIE	S_WITHIN_LA	BORATORY_C	ONTROL
QC Limits* (Aqueous)				
QC Limits* (Solid)	70to_130	to	to	
LL_to_UL	to	to	to	

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 70% or more than 130%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:

- (1) Obvious interference is present on the chromatogram (e.g., unresolved complex mixture);
- (2) Percent moisture of associated soil/sediment sample is >25% and surrogate recovery is >10%; or
- (3) The surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

All criteria were metX
Criteria were not met and/or see below

# VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- Matrix duplicate Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 70 130% of the true value. Lower recoveries of n-nonane are permissible (if included in the calibration of the C9-C12 aliphatic range), but must be noted in the narrative if <30%.</p>

# 

Note: Outside control limits due to high level in sample relative to spike amount. No action taken, MS/MSD results apply only to the unspiked sample. Unspiked sample from another project.

All criteria were metX
Criteria were not met and/or see below

No action is taken on MS/MSD results alone to qualify the entire case. However, used informed professional judgment, the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, the qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that the laboratory is having a systematic problem in the analysis of one or more analytes, which affects the associated samples.

# 2. MS/MSD – Unspiked Compounds

List the concentrations of the unspiked compounds and determine the % RSDs of these compounds in the unspiked sample, matrix spike, and matrix spike duplicate.

	CONCENTR				
COMPOUND	SAMPLE	MS	MSD	%RPD	ACTION
	14 - 1894 - 28				
				Re di di	

Criteria: None specified, use %RSD ≤ 50 as professional judgment.

#### Actions:

If the % RSD > 50, qualify the results in the spiked sample as estimate (J). If the % RSD is not calculable (NC) due to nondetect value in the sample, MS, and/or MSD, use professional judgment to qualify sample data.

A separate worksheet should be used for each MS/MSD pair.

All criteria were met _ met and/or see below	_X

# VIII. LABORATORY CONTROL SAMPLE (LCS/LCSD) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

LCS Recoveries Criteria

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT	ACTION	
LCS_RE	COVERY_WITHIN_L	ABORATORY	Y_CONTROL_LIM	TS	
		<u> </u>			

### Criteria:

- Refer to QAPP for specific criteria.
- \* The spike recovery must be between 70% and 130%. Lower recoveries of n-nonane are permissible (if included in the calibration of the C9-C12 aliphatic range). If the recovery of n-nonane is <30%, note the nonconformance in the executive narrative.

#### Actions:

Actions on LCS recovery should be based on both the number of compounds that are outside the %R criteria and the magnitude of the excedance of the criteria.

If the %R of the analyte is > UL, qualify all positive results (j) for the affected analyte in the associated samples and accept nondetects.

If the %R of the analyte is < LL, qualify all positive results (j) and reject (R) nondetects for the affected analyte in the associated samples.

If more than half the compounds in the LCS are not within the required recovery criteria, qualify all positive results as (J) and reject nondetects (R) for all target analyte(s) in the associated samples.

# 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix (1 per 20 samples per matrix)? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected. Discuss the actions below:

		Crit	All criteria eria were not met and		metN/A e below
IX. FIELD/LA	BORATOR	Y DUPLICATE PR	ECISION		
Sample IDs:		<del>-</del>	^	/latrix:_	
Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measures only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.					
COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
No field/laborator used to assess pr	y duplicate ecision. RI	analyzed with this PD within laborator	data package. MS/Ny and generally acce	SD rec	overies RPD control limits.
Criteria:  The project QAPP should be reviewed for project-specific information.  RPD ± 30% for aqueous samples, RPD ± 50 % for solid samples if results are > SQL.					
If both samples an	d duplicate	e are <5 SQL, the f	RPD criteria is double	esuits a ed.	ire <u>&gt;</u> SQL,
SQL = soil quantitation limit					
Actions:					
If both the sampl calculable (NC). N	e and the o action is	duplicate results needed.	are nondetects (N	D), the	RPD is not
Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria.					

If one sample result is not detected and the other is  $\geq 5x$  the SQL qualify (J/UJ).

judgment to determine if qualification is appropriate.

judgment to determine if qualification is appropriate.

Note: If SQLs for the sample and duplicate are significantly different, use professional

If one sample value is not detected and the other is < 5x the SQL, use professional

All criteria were met	X
Criteria were not met and/or see below	

# XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TICs).

- Verify that the target analytes were within the retention time windows.
  - Retention time windows must be re-established for each Target VPH
     Analyte each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.
  - o Coelution of the m- and p- xylene isomers is permissible.
  - o All surrogates must be adequately resolved from individual Target Analytes included in the VPH Component Standard.
  - For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
  - The n-pentane (C5) and MtBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.

Note: Target analytes were within the retention time window.

2. If target analytes and/or TICs were not correctly identified, request that the laboratory resubmit the corrected data.

All criteria were met _	_X
Criteria were not met and/or see below	

#### XII. QUANTITATION LIMITS AND SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

In the space below, please show a minimum of one sample calculation:

MC45834-3MS

VPH (C7 – C10 Aliphatics) RF =  $4.015 \times 10^5$ 

FID

, i i \*

 $[] = (86097226)/(4.015 \times 10^5)$ 

[] = 214.4 ppb Ok

MC45838-1MS

VPH (C9 – C10 Aromatics) RF =  $9.58 \times 10^5$ 

PID

 $[] = (331862372)/(9.58 \times 10^5)$ 

[] = 346.4 ppb Ok

- If requested, verify that the results were above the laboratory method detection 2. limit (MDLs).
- 3. If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION

If dilution estimate r	was results	not (J) f	performed or the affect	and ted c	the omp	results ounds. I	were List the	above affecte	the ed sa	concentra mples/con	ition npou	range, ınds:
			<del> </del>		<u> </u>					<u>.</u>		

#### **EXECUTIVE NARRATIVE**

SDG No:

MC45838

Laboratory:

**Accutest, Massachusetts** 

Analysis:

MADEP EPH

Number of Samples:

. .

Location:

BMSMC, Building 5 Area

Humacao, PR

**SUMMARY:** 

Five (5) samples were analyzed for Extractable TPHC Ranges by method MADEP EPH. Samples were validated following the METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

**Critical issues:** 

None

Major:

None

Minor:

None

**Critical findings:** 

None

Major findings:

None

**Minor findings:** 

None

COMMENTS:

Results are valid and can be used for decision making purposes.

Reviewers Name:

Rafael Infante

Chemist License 1888

Signature:

Date:

May 31, 2016

# SAMPLE ORGANIC DATA SAMPLE SUMMARY

Sample ID: MC45838-1A

Sample location: BMSMC Building 5 Area

Sampling date: 5/9/2016 Matrix: Groundwater

METHOD: MADEP EPH

Analyte Name	Result	Units Di	lution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	100	ug/L	1	-	U	Yes
Ç9 - C18 Aliphatics	100	ug/L	1	-	U	Yes
Ç19 - C36 Aliphatics	100	ug/L	1	-	U	Yes
Ç11 - C22 Aromatics	100	ug/L	1	-	U	Yes

Sample ID: MC45838-3A

Sample location: BMSMC Building 5 Area

Sampling date: 5/10/2016

Matrix: Groundwater

METHOD: MADEP EPH

Analyte Name	Result	Units Di	ilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	110	ug/L	1	-	IJ	Yes
Ç9 - C18 Aliphatics	110	ug/L	1	-	υ	Yes
Ç19 - C36 Aliphatics	110	ug/L	1	-	U	Yes
Ç11 - C22 Aromatics	110	ug/L	1	•	U	Yes

Sample ID: MC45838-4A

Sample location: BMSMC Building 5 Area

Sampling date: 5/10/2016

Matrix: Groundwater

METHOD: MADEP EPH

Analyte Name	Result	Units [	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	110	ug/L	1	•	U	Yes
Ç9 - C18 Aliphatics	110	ug/L	1	-	υ	Yes
Ç19 - C36 Aliphatics	110	ug/L	1	-	U	Yes
C11 - C22 Aromatics	110	ue/L	1	-	11	Ves

# **DATA REVIEW WORKSHEETS**

type of validation	Limited:	Date:5/0 Shipping date:	r:_MC45838 9-10/2016 05/10/2016 2	
REVIEW OF EXT	RACTABLE PETROL	EUM HYDROCA	RBON (EPHs) PAC	KAGE
validation actions. This more informed decision were assessed according precedence METHOD HYDROCARBONS (VF (2004). Also the gener Support Section. The Common section is actionally section.	es for evaluating volate document will assist the nand in better serving ing to the data validation FOR THE DETERION, Massachusetts Depral validation guidelines CC criteria and data validation describes and d	e reviewer in using the needs of the on guidance docu MINATION OF artment of Enviror promulgated by lation actions lister	professional judgme data users. The sam ments in the followin EXTRACTABLE PEnmental Protection, Rothe USEPA Hazardord on the data review was professional protection.	nt to make ple results g order of TROLEUM evision 1.1 us Wastes
The hardcopied (labo received has been review for SVOCs included)	ratory name) _Accuted ewed and the quality cou ded:	st_Laboratories ntrol and performa	data nce data summarized	package . The data
Trip blank No.:	MC45838 _5  	<u> </u>	c:_Groundwater/Soil_	
X Data Complet X Holding Times N/A GC/MS Tuning N/A Internal Stand X Blanks X Surrogate Rec X Matrix Spike/M	s g ard Performance coveries	X Field Du X Calibrati	ons nd Identifications nd Quantitation	
Overall _Extractable_Petroleum (C9_to_C36_Aliphatics;	n_Hydrocarbons_by_GC _C11_to_C22_(Aromati	_by_Method_MAC	EP EPH. REV 1.1.	comments:
Definition of Qualifiers:				
J- Estimated resul J- Compound not of R- Rejected data JJ- Estimated nond Reviewer:	detected			

		Chiena were not in	let and/or see below
l.	DATA COMPLETNE A. Data Packag		
MISSIN	NG INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
_			
3.	Other		Discrepancies:

All criteria were met \_\_x\_\_

All criteria were metX
Criteria were not met and/or see below

#### **HOLDING TIMES**

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED	DATE ANALYZED	ACTION
Samples	extracted and a	nalyzed within me	thod recommended	d holding time
	<del>-</del>			

# Criteria

# Preservation:

Aqueous samples must be acidified to a pH of 2.0 or less at the time of collection.

Soil samples must be cooled at 4 ± 2 °C immediately after collection.

### Holding times:

Samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

Cooler temperature	e (Criteria: 4 <u>+</u> 2 °C):_	4.8°C
--------------------	---------------------------------	-------

Actions: Qualify positive results/nondetects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ). If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R). If samples were not at the proper temperature (> 10°C) or improperly preserved, use professional judgment to qualify the results.

	All criteria were metX Criteria were not met and/or see below								
CALIBRAT	CALIBRATIONS VERIFICATION								
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.									
Dat	e of initial calib	ration:02/04	V16						
Dat	es of initial cali	bration verification:_	02/04/13						
ins	rument ID num	bers:GCD	E						
Mat	Matrix/Level:AQUEOUS/MEDIUM_								
DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED					
Initial cali	bration and init	ial calibration verifica	ation meet method spe	cific requirements.					

#### Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be
  equal to or less than 25% over the working range for the analyte of interest.
  When this condition is met, linearity through the origin may be assumed, and the
  average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of interest. Calculate the collective CFs for C9-C18 Aliphatic Hydrocarbons, C19-C36 Aliphatic Hydrocarbons, and C11-C22 Aromatic Hydrocarbons using the FID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest.
  - o The area for the surrogates must be subtracted from the area summation of the range in which they elute.
  - The areas associated with naphthalene and 2-methylnaphthalene in the aliphatic range standard must be subtracted from the uncorrected collective C9-C18 Aliphatic Hydrocarbon range area prior to calculating the CF.

#### DATA REVIEW WORKSHEETS

#### Criteria- CCAL

- At a minimum, the working calibration factor must be verified on each working day, after every 20 samples or every 24 hours (whichever is more frequent), and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.
- If the percent difference (%D) for any analyte varies from the predicted response by more than ±25%, a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects.

If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

## CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:02/04/16	<u> </u>
Dates of continuing calibration verification:	_05/13/16;_05/16/16
Dates of final calibration verification:	_05/13/16;_05/16/16
Instrument ID numbers:GCDE	
Matrix/Level:_SOIL/AQUEOUS/MEDIUM	

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
Continu	uing and ending	calibration verificati	on meet method speci	fic requirements.

A separate worksheet should be filled for each initial curve

			Criteria were not	All criteria were met met and/or see below _	X
				met and/or see below	
V A. BLANI	K ANALYSIS F	RESULTS (Se	ections 1 & 2)		
magnitude of blanks associ problems with evaluated to case, or if the Method Blank	contamination ated with the s any blanks determine whe problem is ar	problems. The samples, included in the control of t	ne criteria for evaluding trip, equipma associated with ere is an inherencurrence not affects suspected of I	etermine the existence luation of blanks apply or nent, and laboratory blan the case must be care t variability in the data for ting other data. A Laborating highly contaminate	nly to ks. If efully or the atory
List the conta separately.	mination in the	e blanks belo	w. High and low l	evels blanks must be tre	ated
Laboratory bla	anks				
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS	
				ITERIA	
Field/Trip/Equi	ipment				
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS	
_NO_TRIP/FIE _DATA_PACK	'A O.E.	NT_BLANKS		SOCIATED_WITH_THIS	
					_

All criteria were met _	_X
Criteria were not met and/or see below	

## V B. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is < sample quantitation limit (SQL) and < AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but < AL, report the compound as not detected (U) at the reported concentration.

If the concentration is > AL, report the concentration unqualified.

CAMPLEID

All criteria were metX
Criteria were not met and/or see below

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery. Matrix: solid/aqueous

SUPPOSATE COMPOUND

SAMPLEID	SURRC S1	GATE COMPO S2	OUND S3	S4	ACTION
_SURROGATE					ORY_CONTROL
S1 = o-Terpher	nyl 40-14(	1%	S2 = 2-FI	uorobiphenyl	40-140%
S3 = 1-Chloroo				omonaphthale	
QC Limits (%)*					
_LL_to_UL_ QC Limits* (Sol	_40_to_14 id)	040_to_140	40_to_	14040_to	_140_
_LL_to_UL_	to	to	to	to	

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 40% or more than 140%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:

- (1) Obvious interference is present on the chromatogram (e.g., unresolved complex mixture);
- (2) The surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

Crite	All criteria were metX eria were not met and/or see below
VII. A MATRIX SPIKE/MATRIX SPIKE DUPLIC	
This data is generated to determine long term	precision and accuracy in the analytical

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- Matrix duplicate Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 40 140% of the true value. Lower recoveries of n-nonane are permissible but must be noted in the narrative if <30%.</p>

MS/MSD Recoveries and Precision Criteria							
Sample ID:			Matrix/Level:				
List the %Rs, RF	PD of the compounds wh	ich do no	t meet t	he QC criteria.			
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION		
<u></u>					<del></del>		
7		-					
<u> </u>			995				

duplicate used to assess accuracy.

Note: No MS/MSD analyzed. No action taken, blank spike/blank spike

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All criteria were met	X
Criteria were not met and/or see below	

No action is taken on MS/MSD results alone to qualify the entire case. However, used informed professional judgment, the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, the qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that the laboratory is having a systematic problem in the analysis of one or more analytes, which affects the associated samples.

## 2. MS/MSD - Unspiked Compounds

List the concentrations of the unspiked compounds and determine the % RSDs of these compounds in the unspiked sample, matrix spike, and matrix spike duplicate.

	CONCENTR				
COMPOUND	SAMPLE	MS	MSD	%RPD	ACTION
				<u> </u>	<del></del>

Criteria: None specified, use %RSD ≤ 50 as professional judgment.

## Actions:

If the % RSD > 50, qualify the results in the spiked sample as estimate (J). If the % RSD is not calculable (NC) due to nondetect value in the sample, MS, and/or MSD, use professional judgment to qualify sample data.

A separate worksheet should be used for each MS/MSD pair.

			Criteria	All were not me	criteria were metX et and/or see below
	VIII.	LABORATORY CON	TROL SAMPLI	E (LCS/LCSI	D) ANALYSIS
matric	This d	lata is generated to de	termine accura	cy of the ana	alytical method for various
	1.	LCS Recoveries Crite	eria		
		List the %R of compo	ounds which do	not meet the	e criteria
LCS II	D	COMPOUND	% R	QC LIMIT	ACTION
_LCS	S_REC	OVERY_WITHIN_LAB	ORATORY_CO	ONTROL_LIN	MTS
	Action Action that ar	Refer to QAPP for sp The spike recovery m n-nonane are permis nonconformance in must be < 25%.  s: s on LCS recovery sh re outside the %R and	ust be between sible. If the rec the executive nould be based	covery of n-n narrative. R	40%. Lower recoveries of sonane is <30%, note the PD between LCS/LCSD e number of compounds itude of the excedance of
he ass f the ' for the f more qualify	sociated %R of to affected than h	d samples and accept of the analyte is < LL, que described analyte in the assocional the compounds in the compounds in the results as (J) and	nondetects. alify all positive ated samples. he LCS are no	e results (j) a	or the affected analyte in and reject (R) nondetects required recovery criteria, all target analyte(s) in the
2.	Freque	ency Criteria:			
er ma f no, t he eff	itrix)? <u>Y</u> he data ect and	<u>es</u> or No. ⊢may be affected. Use	professional j	udgment to	matrix (1 per 20 samples determine the severity of slow and list the samples

		Crit	All crite eria were not met an		e metX below		
IX. FIELD/LA	BORATOR	Y DUPLICATE PR	ECISION				
Sample IDs:				/latrix:			
overall precision. results may have laboratory perform variance than was	Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measures only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.						
COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION		
No field/laboratory RPD used to ass	duplicate ess precisi	analyzed with this on. RPD within lab limits	data package. BS/B: oratory and generall	SD recor y accept	veries results table control		
	<u> </u>						
Criteria: The project QAPP should be reviewed for project-specific information. RPD $\pm$ 30% for aqueous samples, RPD $\pm$ 50 % for solid samples if results are $\geq$ SQL. If both samples and duplicate are $<$ 5 SQL, the RPD criteria is doubled.							
SQL = soil quantita	ation limit						
Actions:							
If both the sample calculable (NC). No	If both the sample and the duplicate results are nondetects (ND), the RPD is not calculable (NC). No action is needed.						
Qualify as estima exceeded the above	ted positiv ve criteria.	e results (J) and	nondetects (UJ) for	the co	mpound that		
If one sample resu	It is not de	tected and the other	er is $\geq 5x$ the SQL qu	alify (J/l	JJ).		

If one sample value is not detected and the other is < 5x the SQL, use professional judgment to determine if qualification is appropriate.

Note: If SQLs for the sample and duplicate are significantly different, use professional

judgment to determine if qualification is appropriate.

All criteria were met	x
Criteria were not met and/or see below	

## XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TICs).

- 1. Verify that the target analytes were within the retention time windows.
  - Retention time windows must be re-established for each Target EPH
     Analyte each time a new GC column is installed, and must be verified
     and/or adjusted on a daily basis.
  - o The n-nonane (n-C9) peak must be adequately resolved from the solvent front of the chromatographic run.
  - o All surrogates must be adequately resolved from the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.
  - For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
  - The n-pentane (C5) and MtBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.
- 1a. Aliphatic hydrocarbons range:
  - o Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for n-C9 and 0.01 minutes before the Rt for n-C19.
  - Determine the total area count for all peaks eluting 0.01 minutes before the Rt for n-C19 and 0.1 minutes after the Rt for n-C36.

Are the aliphatic hydrocarbons range properly determined?

Yes? or No?

Comments:

- 1b. Aromatic hydrocarbons range:
  - Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for naphthalene and 0.1 minutes after the Rt for benzo(g,h,i)perylene.
  - Determine the peak area count for the sample surrogate (OTP) and fractionation surrogate(s). Subtract these values from the collective area count value.

Are the aliphatic hydrocarbons range properly determined?

Yes? or No?

Comments:

		Criteri	a were not	All criteria met and/or	were met see belov	X
2.	If target analytes a laboratory resubmit t	nd/or TICs were no he corrected data.	t correctly	identified,	request t	that the
3.	evaluated for potenti % recovery of the fra basis by quantifying and aromatic fractio naphthalene or 2-m the total concentrations	mination - Each sar al breakthrough on a actionation surrogate naphthalene and 2-r ns of the LCS and l ethylnaphthalene in tion for naphthalene ion must be repeate	sample sp (2-bromor nethylnaph -CSD. If e the alipha or 2-met	ecific basis naphthalene nthalene in either the catic fraction hylnaphtha	by evaluate) and on both the aconcentran exceeds	iting the a batch aliphatic tion of 5% of he LCS
	NOTE:	The total concernethylnaphthalend summation of the aliphatic fraction a aromatic fraction.	in the LO	CS/LCSD pointration d	air includetected	les the
	Comments:Concel_concentration_for_n	ntration_in_the_alipha aphthalene_and_2-m	atic_fraction	thalene		
4.	Fractionation Chec containing 14 alkane each constituent. The fractionation efficience optimum hexane volunot allowing significationation in the fractionation of the fractional of	es and 17 PAHs at a e Fractionation Check by of each new lot of time required to efficient ant aromatic hydroca ctionation check solu	nominal c Solution n silica gel/c ently elute a arbon brea tion, exclu-	check solution oncentration nust be use cartridges, a aliphatic hy kthrough. Finding n-non-	n of 200 red to evaluand estable drocarbon For each anne, the F	repared ng/µl of the the lish the swhile analyte
	Is a fractionation che	ck standard analyzed	?		Yes? or	No?
	Comments: Not applie	cable.				

All criteria were metX_	
Criteria were not met and/or see below	

## XII. QUANTITATION LIMITS AND SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

In order to demonstrate the absence of aliphatic mass discrimination, the response ratio of C28 to C20 must be at least 0.85. If <0.85, this nonconformance must be noted in the laboratory case narrative.

The chromatograms of Continuing Calibration Standards for aromatics must be reviewed to ensure that there are no obvious signs of mass discrimination.

Is aliphatic mass discrimination observed in the sample?

Yes? or No?

Is aromatic mass discrimination observed in the sample?

Yes? or No?

1. In the space below, please show a minimum of one sample calculation:

Blank Spike Dup.

EPH (C11 – C22, Aromatics)

RF = 98200

[] = (40272130)/(98200)

[] = 410 ppb Ok

Blank Spike Dup.

EPH (C19 - C36, Aliphatics)

RF = 66810

[] = (766074)/(66810)

[] = 11.5 ppb Ok

# **DATA REVIEW WORKSHEETS**

- 2. If requested, verify that the results were above the laboratory method detection limit (MDLs).
- 3. If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION
		<del>-</del>

If dilution was not performed, affected samples/compounds:	results	(J) fo	r the	affected	compounds.	List the
	 					_